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A multivariate SIMCA index as discriminant in wood pellet quality assessment

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ABSTRACT

The pellet market has experienced a continuous development and increase in recent years due to a number of positive properties of this enhanced biomass. However the supply chain has not been entirely able to follow the same trend, causing some issues, often related to the quality of traded products. These problems can be partially solved by ensuring a continuous and reliable flow of information regarding the quality parameters of wood pellets from the producers to the final users. The aim of this work is to define a metric index for quality parameters that can detect the certifiability of analyzed samples compared with those on the market. The model is built on measured quality parameters of certified and non-certified wood pellet samples taken from products on the market applying a multivariate class modelling methodology (soft independent modelling of class analogy, SIMCA). Results showed that the model can predict the general quality of some test samples and that its precision, already fairly high, can be constantly improved by adding new model samples. The output of the model is also the relative influence (modelling power) of each variable in the prediction of certifiability. The SIMCA model could be easily integrated and implemented on the most common digital platforms where users (private, laboratories, agencies, etc.) could test their samples and verify if the index of their pellet falls within the area defined by the model for certified samples.

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1. Introduction

The increased global demand for energy is forcing the renewable sector to find new strategies and supply solutions. The demand for renewable energy sources has continued to rise, and data available for 2011 and 2012, published in EU reports, give an estimated supply of final renewable energy of about 19% share among others [1].

This has also affected the biomass trade with specific regard to wood-based products as substitutes for fossil fuels. Biomass feedstock is one of the more important strategies since its share among renewable energy sources is growing.

The production and use of traditional or technologically improved woody biofuels can contribute to the overall greenhouse gases (GHG) reduction [2] due to their close-to-neutral carbon emission [3]. This has strongly encouraged many European countries to set up subsidy schemes for the substitution of oil, coal and gas residential and industrial heating systems or power plants by others run on biomaterials.

Among these products, the use of wood pellets as a biofuel is rapidly increasing and is expected to grow more than 10 fold by the year 2020 [4]. In the period 2009–2012 the production of pellets in the EU has increased by 30%, reaching 9.2 million tons in 2010 and 10.6 million tons in 2012 [5]. More than half of this production is concentrated in few core producing areas mainly located in

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Abbreviations: AbTr, Norway spruce whole tree; AbSt, Norway spruce whole stem; AbDe, Norway spruce debarked stem; A_d, ash content on dry basis; BD_{ar}, bulk density as received; CCP, critical control point; CV, cross-validates; DE_{ar}, solid density as received; DU_{ar}, mechanical durability as received; EN, European norms; FaSt, beech whole stem; FaBr, beech branches; GAS, general analysis sample; HHV_{ar}, high heating value as received; HHV_{0af}, dry and ash free high heating value; LHV_{ar}, low heating value as received; M_{ar}, moisture content as received; PCA, principal component analysis; SIMCA, soft independent modelling of class analogy; UNI, Italian standardization body; VCS, virtual certified sample.

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Northern and Central Europe [6]. EU wood pellets consumption increased to 11 million tons in 2010 [7]. This gap has caused a shortage of pellets on the European market, which has resulted in increasing prices and a dependence on importations [8] from USA, Canada, South America, New Zealand and Russia [9]. These data prove that the EU currently represents the most important market for wood pellets since they constitute an effective alternative to other traditional energy fossil sources, the prices of which keep increasing per energy unit. Moreover the level of technology required to exploit wood pellets is still lower and less expensive if compared with other renewable sources.

The physical and mechanical properties of pellets play a key role in their diffusion, since more energy can be compacted and stored in the same amount of volume making their storage costs lower compared with other traditional wood fuels [10,11]. The regular geometrics and steady energy characteristics [10] allow pellets to be handled, stored and used in automatic appliances [12,13] equipped with mechanical or pneumatic conveyors.

Wood pellets quality is affected by a number of parameters linked to the: *i.* feedstock properties following the general rules *what goes in, comes out*; *ii.* feedstock pre-treatment such as comminution before storage, seasoning condition and drying technology; *iii.* production settings; *iv.* post-treatment.

The selection of proper raw materials affects pellets quality, as their properties and constituents are almost unchanged in the final product. By-products from the wood processing industry are the traditional raw material, but their availability has already reached its maximum limit [14]. Alternative raw materials are thus needed and their utilization has to be tested to allow economic and quality productions. Storage conditions, such as drying temperature and natural seasoning, affect the mechanical compaction behavior of sawdust for providing more durable pellets [15]. The optimal moisture content of feedstock should be defined and ensured through precise control and adjustment of its level during the production stage [19]. This is because the moisture contained in the feedstock as bound water is a key player in the formation of inter-particle linkages when its values are maintained between 8% and 13% [15,16].

The pelletizing process is strongly affected by the tree species used, the characteristics of which are then reflected in the final pellet quality [16,17]. The pelletizing pressure, the hold time of pellets in the die channel (i.e.: compaction ratio) and the extruding temperature are directly related to feedstock characteristics [18].

Delivery and handling chains are equally important for maintaining the initial quality level and allowing the proper and widespread use of pellets for energy production that can compete with other bioenergy sources [14].

The fast growth and evolution of the pellet sector in Europe has encouraged the development of specific standards and regulations in the attempt to set some baselines for production, storage and distribution. The standardization process has resulted, by the end of 2012, in the publication of some harmonized European norms (EN) regulating pellets quality requirements. The EN 14961 [20], in part 2, sets the parameter limits to meet different quality classes (A1, A2 and B), while EN 15234-2 [21] refers specifically to pellet fuel quality assurance. The general objective of quality assurance is to guarantee the overall pellet quality level throughout the entire supply chain by maintaining a control system with reliable records at every critical control point (CCP) in the chain. The European standard, officially adopted by the Italian Standardization Body (UNI), is appropriate for the national market that is mainly based on residential appliances, such as stoves and small boilers [22], which are sensitive to the variability of quality parameters.

This paper proposes a multivariate class modelling approach that could extract an index from the measured quality parameters of a wood pellet population randomly purchased from the products available on the market. The index calculated can be set as a baseline for the real quality of the analyzed pellet samples on the market and acts as a threshold in the evaluation of newly analyzed samples. The soft independent modelling of class analogy (SIMCA) is built on the actual properties of certified pellet samples (reference population), and is able to predict the position of a test sample (SIMCA distance) on a scale, defining its potential certifiability referred to the critical distance previously calculated via SIMCA.

2. Materials and methods

2.1. Pellets characterization

2.1.1. Commercial pellets sampling

The sampling campaign lasted for 6 months, during which a total number of 51 pellet bags (15 kg each) were randomly obtained from 25 producers and retailers operating in the Italian market regardless of their provenance, cost or commercial name. The suppliers were not informed about the research to avoid any influence on the sample choice from the original batch prior to delivery. This procedure was established to ensure that all the production parameters affecting the final pellet quality and the selection criteria would not be modified or influenced by the supplier due to the awareness of the aim of the study. All information about the origin, source and raw material used, certification and measured parameters for pellet samples was recorded. Some information was retrieved from the producer or importer when reachable. When this was not possible only the information reported on the packaging was considered.

Nine samples were certified by national standards certification schemes (DINplus, PelletGold), thirteen by the European standard (ENplus), while the others had no certification. For some of them the quality parameters were stated on the bag, while the quality characteristics of others were completely undeclared. Those with stated values were divided into two groups: ones with precise data records and ones with just declared ranges of values.

The sampling was conducted directly by trained Biofuel Laboratory staff from Padova University.

2.1.2. Pellet manufacturing

Five reference feedstock materials were also collected, sorted and pre-dried. They were chosen considering the economic potential of small trees from thinning operations and residues from final felling. Two species were used: Norway spruce (*Picea abies* Karst) and beech (*Fagus sylvatica* L). For the former, whole tree (AbTr), whole stem (AbSt) and debarked stem (AbDe) were collected; for the latter whole stem (FaSt) and branches (FaBr). After grinding in a hammer mill (sieve apertures 6 mm), the moisture content was normalized (12%) and each material was pelletized using an industrial pilot plant (ring die aperture 6 mm), which reproduced the average production conditions available. The pellets produced were sampled and analyzed for the scope of this work as they had been processed under the same conditions of feedstock pre-treatment and seasoning, production settings and post-treatment. The specific moisture state at which each variable was considered refers to the value measured directly on the sample (*as received*) that includes the moisture level within the wood. Only the ash content was referred to *dry basis* since its determination implies the complete evaporation of water.

2.1.3. Pellet analysis

The samples were sent to the Biofuel Laboratory of Padova University for further analyses. On arrival in the Laboratory all samples were managed following EN 14778 [23] and EN 14780 [24] for sampling reduction and test sample preparation. To do this all samples were transferred to anonymous plastic containers and marked with a unique ID number to make them recognizable later in the study.

The quality analyses were based on the classes proposed by standard EN 14961–2 [20]. It delegates the role of specifying the methodology and procedures to determine quality parameters to other satellite norms.

The general analytical procedure consisted of the following tests: moisture content, fines content, mechanical durability, bulk density, dimensions, particle density, ash content and calorific value.

The moisture content (M) on a wet basis was determined following EN 14774–1 [25], placing three subsamples of each pellet type in a ventilated oven at 105 °C until they reached a constant in weight. The quantities, dimension and shape of containers were consistent with the standard.

The fines content was determined on the whole content of the bag by weighing and sieving as stated in EN 15210 [26].

The amount of dust and fines (F) is correlated with the mechanical properties of the pellet with its tendency to disintegrate under stress. This variable was discarded from the statistical analysis since the content of fines in the bag is strictly related to the specific treatment that the bag underwent (*i.e.*: transportation, storage, handling).

The mechanical durability (DU) test was performed on three repetitions of 500 g subsamples using a tumbling machine, the dimensions and speed of which comply with the standard requirements. The percentage of pellets remaining integral compared to the initial subsample weight gives the DU value.

The bulk density (BD) was determined by means of a steel container [27]. The measuring container has a filling volume of 5 l. The final net weight of the test sample referred to the volume of a cubic meter gives the bulk density value. Results are reported as the average of three replications.

The dimensions were measured using a caliper to record the length in mm of a 80 g subsample, selected from the whole pellet batch as specified in standard EN 16127 [28]. In order to determine the diameter class (Ø), a minimum of 10 pellets were selected, their diameters measured twice in perpendicular positions and averaged. Pellets with a length (L) greater than the requirement were hand sorted and measured separately.

The density (DE) of the single pellets was determined by the relationship between the mass of a particle and its volume [29]. The volume was measured *via* the buoyancy in a liquid. The apparent loss in weight between a measurement in air and a subsequent measurement in liquid marks its buoyancy. The volume of the used pellets was calculated given the density of the immersion liquid at controlled room temperature (20 °C). This methodology could be subjected to errors, mainly caused by the rapid uptake of water from pellets. Despite this it has been proved to be more reliable than hydrostatic or stereometric procedures [30]. Density was determined on 40 pellets in 10 replications. This parameter influences the energy density of pellets, as it results from the combination of heating value and solid density.

A representative subsample was ground for further analysis using a laboratory cutting mill to reduce the particle size distribution to a nominal top size of 1 mm required for the General Analysis Sample (GAS).

The ash content (A) was determined on the GAS following the EN 14775 [31] weighing a portion of GAS before and after its complete combustion under fixed temperature conditions until it reached a constant weight. Results are reported as the average of three replications on a wet basis (as received) given the moisture content of the sample being incinerated.

The calorific value was determined according to EN 14918 [32] using the IKA C-200 calorimeter that gives the high heating value as received (HHV_{ar}). To determine it a weighed portion of the GAS was burnt in a high-pressure oxygen bomb under specified conditions. The effective heat capacity of the calorimeter was determined in calibration experiments by combustion of certified benzoic acid under similar conditions. Through the discounting of the moisture and ash content from the initial sample weight, the HHV_{0af} can be retrieved. Results are reported as the average of three replications. For further calculations the HHV_{ar} was converted into LHV_{ar}, which expresses the real energy that can be generally exploited in a domestic burning appliance accounting for the heat expenses due to evaporation of water contained in the fuel and produced during combustion.

The quality analysis was related to the declared parameters stated on the package or in the product declaration (EN 15234–2 [21]) by the producer or trader. The presence of any kind of certification seal was checked and the compliance of measured parameters with reference ones was verified.

2.2. Statistical analysis

2.2.1. Multivariate approach

As stated by Ref. [33] multivariate class-modelling techniques answer the general question if an object *O*, stated as class *A*, really belongs to class *A*. This is a typical question in multivariate quality control. On the contrary, the classification techniques assign objects to one of the classes in the problem. Class-modelling techniques calculate the “prediction probability” with a classification threshold for each modelled class. By using a class-modelling approach, it is possible to attribute objects not only to one or more classes but also to none (*i.e.*, in this case, the object is an outlier).

In order to find an index able to assess the general quality of a population of wood pellet samples randomly obtained from products on the Italian market and to define a baseline for quality parameters of the general pellet market, a SIMCA [34–37] was applied on a qualitative variables dataset. Among the analyzed variables, six were chosen to perform the class-modelling approach and the consequent SIMCA quality index (Table 1). These variables were selected from the parameters used to describe pellet performance because they are widely considered indicators of pellet quality. They also are the easiest to determine even in simple applied laboratories, and this is part of the aim of this work. The low cost of their determination, if compared with other more sophisticated analyses (*i.e.*: chemicals, radioactivity, ash melting point) plays a key role in the future implementation and use of the presented model. Moreover the insertion of more variables does not substantially increase the model evaluation capacity.

2.2.2. Model description

The SIMCA model, computed with the software V-Parvus 2010, is used for supervised pattern recognition. It is a collection of PCA (Principal Component Analysis) models (computed by the NIPALS

Table 1
Variables used for the model construction.

Variable	Abbreviation	Unit	Limit value for ENplus A1 class
Moisture content as received	M_{ar}	%	≤10
Mechanical durability as received	DU_{ar}	%	≥97.5
Bulk density as received	BD_{ar}	$kg\ m^{-3}_{bulk}$	≥600
Solid density as received	DE_{ar}	$g\ cm^{-3}$	≥1.2
Ash content on dry basis	A_d	%	≤0.7
Low heating value as received	LHV_{ar}	$MJ\ kg^{-1}$	≥16.5

algorithm), one for each class of dataset (one in this case), after separate category autoscaling. SIMCA cross-validates (CV) the PCA model of each class (training set), splitting the data (evaluation set) into 4 contiguous groups (CV groups). In our case, the modified model with expanded range was used rather than the technique first introduced [38]. The unweighted augmented SIMCA distance was also considered in building the models. For each class, the number of significant components of the inner space was estimated considering five PC (lowest noise found). For each modelled class a critical square distance based on the F-distribution was calculated using 95% confidence interval. The class boundary was also determined according to the confidence limit. In CV, new observations are projected into each PC model and the residual distances calculated. An observation is assigned to the model class when its residual distance from the model is below the statistical limit for the class. SIMCA allows both the classification and modelling analysis. For classification, all the observations have to be attributed to one of the pre-defined classes. The efficiency was indicated by classification (training set) and prediction (evaluation set) matrices, which reported the percentage of correct classification for each considered class and total percentage ability. The observations for each class, classified outside the model, were also reported. SIMCA expressed the statistical parameters indicating the modelling efficiency. In fact, the observation may be found to belong to multiple classes or not fit any of them (outlier). Unknown objects could also be either classified into one of the classes or recognized as outliers. The modelling efficiency was indicated by sensitivity and specificity parameters. The sensitivity is the measure of how well the classification test correctly identifies the cases really belonging to the class. The modelling power of each variable, which represents the influence of that variable in the definition of the model, was expressed.

In order to express an index for each pellet sample, square SIMCA distances were linearized converting the values into logarithmic scale and then translating them adding a value of 2.5 (in order to have all positive values).

The SIMCA model was built on a training dataset consisting of 18 out of the 23 certified samples with a measured set of attributes. This partitioning was optimally chosen with the Euclidean distances based on the algorithm of [39] that selects objects without the a priori knowledge of a regression model (*i.e.*, the hypothesis is that a flat distribution of the data is preferable for a regression model). A virtual certified sample (VCS) was added to the population used to build the model. Its properties were set at the minimum–maximum requirements level for a pellet sample to be certified class A1 according to the ENplus scheme [40]. The rest of the samples (34) were used as external test.

3. Results and discussion

The results of the analytical phase are summarized in Table 2, where all the samples quality values are averaged by category: certified samples, non-certified samples, reference samples and VCS. As expected, all the values calculated for the certified samples are better than those of non-certified products.

Table 2
Measured values of the parameters used in model building and grouped by 4 functional categories.

	M_{ar}	DU_{ar}	BD_{ar}	DE_{ar}	A_d	LHV_{ar}
	%	%	kg $m^{-3}{}_{bulk}$	g cm^{-3}	%	MJ kg^{-1}
Certified	6.4	98.9	688	1.29	0.68	17.45
Non-certified	7.4	97.6	648	1.27	1.88	16.77
Reference samples	7.2	97.7	680	1.33	1.30	17.05
VCS (limits for A1 class)	10.0	97.5	600	1.20	0.70	16.50

The statistical analysis reports that the total explained variance of the five SIMCA model principal components is 95.77%.

The adimensional logarithmic translated square critical distance, based on the F-distribution, is 2.88. This identifies the class-boundary of the model below which the distance value of an observation is included in the model, while a higher distance value is rejected. All (100%) the 18 certified samples (including the VCS) used to build the model were included in the SIMCA model (internal sensitivity = 100%). The certified samples used as external test were also included (external sensitivity = 100%).

Among the 34 non-certified samples used as external test, 19 were included in the model. These results are summarized in Fig. 1. None of the certified samples were excluded from the model.

The adimensional logarithmic translated square SIMCA distance (along the *x*-axis in Fig. 1) represents a real metric scale where each sample is positioned. It defines its possible certifiability and locates it in a specific quality level or range. The actual certification schemes are based on a single sample analysis over a period of one year, while the model can be used and updated continuously giving narrower limits to the certification bodies. The innovation is represented by considering the entire dataset, that can be nationally or internationally designed, in a multivariate way with respect to traditional approaches, which generally only consider each single parameter referred to some defined limits (minimum or maximum acceptable values for that specific parameter). The SIMCA distance expresses the inclusion of a test sample in the model or its distance from the model. The critical distance of the computed model is 2.88. Samples with higher distance values could then be considered a proxy of lower quality, and this result is obtained in a multivariate approach. Up to now there has been just one paper in the literature aimed at characterization of the pellet market in Italy based on quality parameters and adopting a multiple correlation approach [41].

The position of samples whose raw materials were agricultural residues or a mixture of non-debarked wood species is negatively affected by A_d. At the same time this variable has the highest modelling power, giving the reason for its weight in the model classification.

The relationship between A_d and the raw material used for the pellet production is confirmed considering the five samples specifically produced for the study. In fact for all of them the ash content exceeded the maximum limit value. Only the ash content for AbDe, which lacks the bark fraction due to the debarking process, respects the maximum ash threshold. However the absence of bark, which acts as binding agent during extrusion [42], lowered the DU_{ar} below the minimum.

The LHV_{ar} was the second major contributor in the exclusion of these samples due to low values. The high heating value of wood is more or less constant with some slight differences related to the content of resins and oils in some species. On the other hand the low heating value is mainly affected by the moisture content of the sample, since some of the energy has to be invested in evaporation. This is the case of the excluded samples, which had a moisture content slightly over the population average.

Apart from one, the BD_{ar} of excluded samples is within the minimum specified requirements.

It can be noted that some samples, whose quality parameters exceed the minimum value for a specified variable, are still accepted by the model. This is explained by taking into consideration that the model is built on the measured quality of pellet samples obtained from the market, so has no direct connection with the empirical threshold of the certification schemes. While the limits imposed by the schemes are fixed and based on some

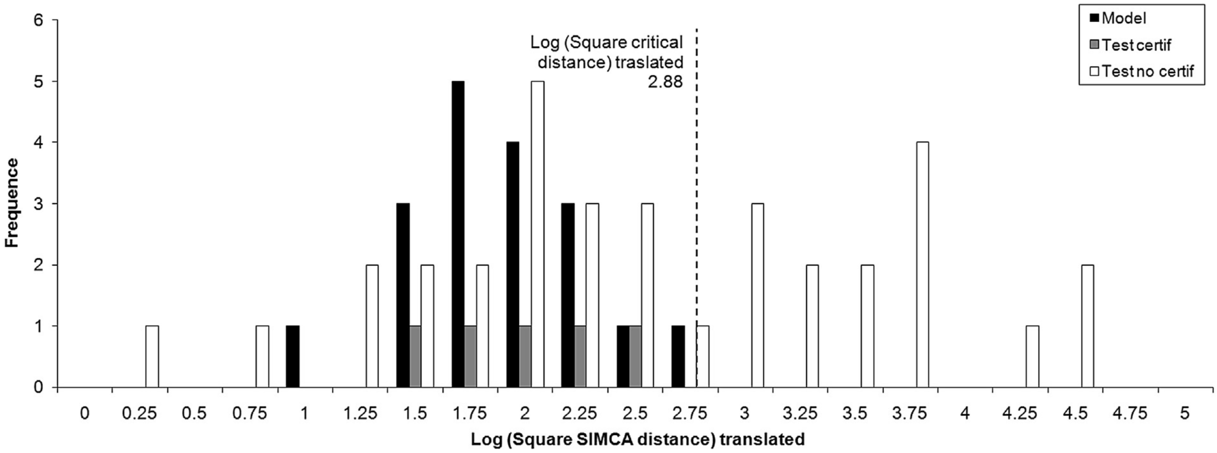

Fig. 1. Number of pellet samples having different logarithmic translated square SIMCA distances from the 18 certified samples used to build the model (*Model*; black), the 5 certified samples used as external test (*Test certif*; Gray) and the 34 non-certified samples used as external test (*Test no certif*; White). The logarithmic translated square critical distance is reported (dashed line).

preliminary studies, the model is adapted and adaptable to the representativeness of the population sampled for this work. The acceptance of all the certified pellets within the model is an indication that the model itself is fully adapted and applicable.

The modelling power values of the six variables are reported in Fig. 2. The power represents the contribution of each variable, together with the others, in the definition of the position of a sample along the *x*-axis. The higher the modelling power score is, the better is the capacity of that variable to explain the position of each sample. The most important variables are A_d, LHV_{ar} and BD_{ar} and this is reflected in the weight that they have in the grading of commercial pellets and the performances related to their use [43]. No power values are below 0.59 (adimensional scale). These variables were mainly responsible for the exclusion of samples from the SIMCA model.

4. Conclusions

The innovative multivariate concept adopted in this study is applied for the construction of a model based on the real quality parameters of wood pellets. This methodology can be spread and applied directly by the market operators to monitor the quality of the measured data and their reaction to improvement actions taken by producers and traders along the chain.

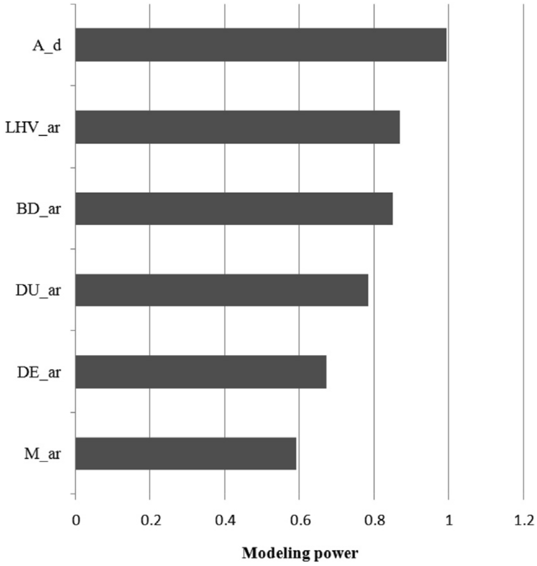

Fig. 2. Variables included in the model ordered by their corresponding contribution to the model definition (modelling power).

Moreover the model, designed to be dynamically updated, can be further improved by adding new samples or other parameters already tested elsewhere [44]. This allows it to be adapted periodically to any quality changes within the market due to raw material variations, seasonal quality fluctuations and importation from other countries. The proposed multivariate SIMCA model could be easily implemented on a web site where web users such as other Italian or European laboratories could test their samples and verify the certifiability of their pellet.

The low number of variables necessary to make the model stable and reliable, together with the low investment required to determine them, are key factors for the application of the model in practice.

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